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To: LX Team
From: Jim Carnahan and Paul Gundlach
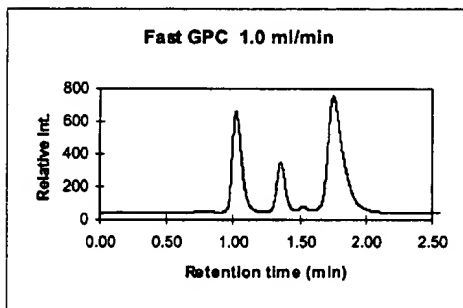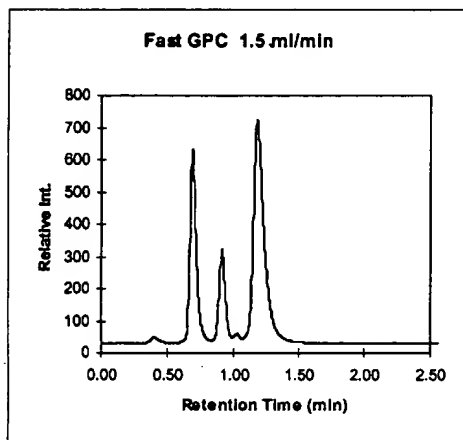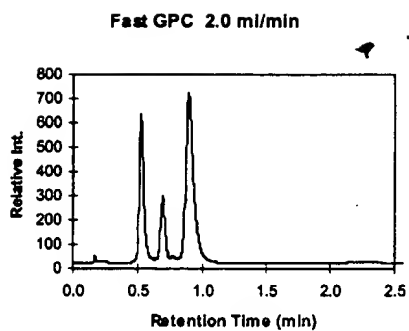Subject: Rapid Screening of Combinatorial LX Polymer Reactions.

Summary: GPC using a short, small pore column permits separation of the reagents BPA and DPC from the polymeric products of LX synthesis in 1.2 minutes at the maximum flow rate tested. The phenol by-product, if present, is convoluted with the BPA peak. A major time delay in a reaction -analysis sequence will be dissolution of the polymer product.

Experimental: GPC was run using a Waters 590 pump, a Waters WISP 712 autosampler and a Waters Model 441 absorbance detector at 254 nm. The column was a PL Gel pre-column 5 μm 100   50 X 7.0 mm part no. 1110-1520. The solvent was chloroform with 1% ethanol at flow rates between 1.0 and 2.0 ml/min. Data collection was through a Waters Assoc. Millenium data system.

Results and Discussion

The intention was to separate the polymer from the reactants to provide rapid measurement of degree of reaction as well as provide a clean polymer fraction for subsequent molecular weight determination. The short column used for this work has a 100   packing and is not capable of separation of polymer by molecular weight.   A series of polycarbonates ranging in MW from 17000 to 102500 showed retention times of 0.532 to 0.527 at the 2.0 ml/min rate and a plot of log MW vs. retention time was not linear, as expected. Examination of an oligomer mixture showed separation from the DPC peak.   The chromatograms for three flow rates for separation of a model system (LX-OQ, DPC and BPA) on the 100   column are shown in Figures 1-3, below. The first large peak is the polymer followed by DPC, a small unknown component and then BPA and phenol. Plotting the traces as a function of retention volume showed no peak separation degradation at the higher flow rate suggesting that we may be able to run at an even higher flow rate. We have not evaluated the effects of Fries products or branched resin on the separation. Radislav Potyrailo and John Lemmon are evaluating the fluorescence spectra of those materials and if they are sufficiently distinguishable from PC, we can add a fluorescence detector after the UV detector and try to quantify the presence of the Fries products as well.

EXHIBIT B

Fast GPC 2.0 ml/min

Relative Int.

Retention Time (min)

Fast GPC 1.5 ml/min

Relative Int.

Retention Time (min)

Fast GPC 1.0 ml/min

Relative Int.

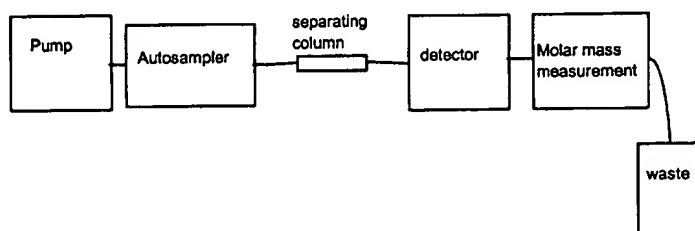Retention time (min)

Measurement of molecular weight.

The column used is not likely capable of useful MW determinations. We have on order a similar sized column packed with a linear packing material that may provide a measure of MW. A better alternative to

trying to measure MW distribution is to add a serial detector for absolute MW. This could be light scattering or differential viscometry with the UV detector signal used for concentration determination. We prefer light scattering because of its mechanical simplicity. Viscotek has a commercial right angle light scattering detector that should be capable and Wyatt Technology offers the Dawn and mini-Dawn multi-angle instruments. We also have proposed a locally built device for in-line or side-stream measurement of average MW in the approximately 250 microliter volume of the polymer elution and are working on a disclosure for that. A block diagram of an in-line system is shown in Figure 4.

Sample preparation. The dissolution of samples from combinatorial synthesis schemes is likely to be the major time delay in the analysis of LX polymerization reactions. If the reactions are carried out in 96 well plates or arrays of vials that after reaction can have solvent added robotically and be subsequently capped, then a shaker or array rotation device could dissolve the polymers prior to analysis. Our current autosamplers can handle individual vials but the 96 well plate format will require purchase of one of the commercial autosamplers configured for that format.

Block Diagrams of Fast GPC Instrumentation Options

Configuration A

Configuration B